Chemistry Unit
Validation of Analytical Procedures

1 Purpose

This document supplements the *FBI Laboratory Practices for Developing Methods and Validating Technical Procedures, FBI Laboratory Practices for Validating Chemical Procedures,* and the *FBI Laboratory Quality Assurance Manual* for the validation of new analytical procedures in the Chemistry Unit (CU).

2 Scope

This document applies to personnel that validate new analytical casework procedures in the CU. Validation starts after a method is acquired and/or developed. If a method needs to be developed in CU (including the modification of an acquired method), the method development will be a planned activity. The method development plan must be recorded, approved by the applicable Technical Leader (TL), and any changes to the plan will be communicated to all personnel involved in the method development.

The validation of an analytical procedure is referred to as a validation study in the CU. The performance characteristics that are evaluated during a validation study will be based on the scope of the analytical procedure. The validation study must be completed, reviewed, and approved prior to the procedure's first use in casework, except as noted within this procedure.

3 Responsibilities

3.1 The Individual/Group performing the method development or validation study will:

- Develop, record, and ensure approval of a method development plan utilizing the CU *Method Development Plan* form (Appendix A). Retain the form.
- Record and/or reference any other technical work relied upon to support the usage of a novel methodology or process.
- Record and retain the results of the method development.
- Develop, record, and ensure approval of a validation plan utilizing the applicable CU *Validation Plan* form (Appendices B-D). Retain the form.
- Record and retain the results of the validation study. The supporting records may include validation data, instrument optimization charts, calculations, relevant literature references, etc.
- Ensure technical review and approval of the validation study utilizing the *Validation of Chemical Procedures Review Form* (7-267) or the CU *Validation*

Plan and Review- Physical Properties Only form (Appendix D), as applicable. Retain the form.
- Complete a *Validation Summary* form (Appendix E). Retain the form.
- Maintain all supporting records related to method development and validation studies conducted within the CU within binders, in electronic format, and/or within case notes, as appropriate. Method development and validation study binders will be stored within the CU's file cabinets and/or bookshelves.

3.2 The applicable TL or Subject Matter Expert (SME) will:

- Review and approve method development plans by signing the CU *Method Development Plan* form. If the TL is the "Lead Scientist" for the method development, then another internal SME (if available) will review and approve the method development plan.
- Review and approve validation plans by signing the applicable CU *Validation Plan* form. If the TL is the "Lead Scientist" for the study, then another internal SME (if available) will review and approve the validation plan.
- Review and approve validation studies by signing the 7-267 form or the CU *Validation Plan and Review- Physical Properties Only* form. If the TL is the "Lead Scientist" for the study, then another internal SME (if available) will sign the CU *Validation Plan and Review- Physical Properties Only* form.
- Review and approve validation summaries by signing the CU *Validation Summary* form. If the TL is the "Lead Scientist" for the study, then another internal SME (if available) will review and approve the validation summary.

3.3 The UC will:

- Review and approve method development plans if there is not another internal SME available.
- Review and approve validation plans by signing the CU *Validation Plan* form.
- Review and approve validation studies by signing the 7-267 form or the CU *Validation Plan and Review- Physical Properties Only* form.
- Review and approve validation summaries by signing the CU *Validation Summary* form.

4 Procedures

4.1 Validation Studies

Validation studies of chemical procedures in the CU will be performed following the requirements outlined in the *FBI Laboratory Practices for Validating Chemical Procedures*. These requirements may be adjusted based on the scope of the procedure and professional judgment (e.g., safety considerations, differences in sample matrices, availability of reference materials).

Validation studies of applicable, non-chemical procedures (i.e., physical property measurements) in the CU will be limited to the characteristic listed in section 4.1.1 of this document.

Validation studies for casework involving the analysis of unknowns will be conducted as detailed in section 4.1.2 of this document.

4.1.1 Performance Characteristic for Measurement of a Physical Property

4.1.1.1 Accuracy

Accuracy is the closeness of an analytical result to its true value and is affected by systematic error (bias) and random error (precision). The accuracy of a physical property measurement can be determined by comparison of that measurement result with the true value. At a minimum, ten measurement replicates of a reference material with a known physical property value are made. The accuracy is calculated as the percent difference of the average measured value from the known value. In most instances, the preferred accuracy is ±15% or less, but larger values may be unavoidable and are acceptable if accompanied by proper justification.

4.1.2 Performance Characteristics for the Analysis of Unknown Component(s)

Due to the nature of unknown component analysis, the validation may be conducted either at the time of analysis or immediately following. Generally, the only performance characteristic that needs to be validated is interferences.[1]

4.2 Record, Review, and Maintain Validation Study Results

Upon completion of the validation study, the UC or appropriate TL will assign appropriate personnel as technical reviewer(s) of the validation study results. If the UC is qualified to do so, he/she may perform the technical review. The technical review must take place before the

[1] In this scenario, the requirements of completing a validation plan, review form, and summary will be waived.

procedure is placed into use.[2] The technical reviewer(s) will complete the 7-267 or the CU *Validation Plan and Review- Physical Properties Only* form. After the technical review is complete, the UC and appropriate TL or SME (as applicable) will review the validation records and record their approval on the 7-267 or the CU *Validation Plan and Review- Physical Properties Only* form. The completed review form will be maintained with the validation study data. Once a new analytical procedure has final approval by the UC, and before it is used in casework, the procedure will be formally written and reviewed following the appropriate Laboratory Division and CU practices.

Validation study records will be maintained within the CU's validation file cabinets, bookshelves, in electronic format, or, with respect to case-specific validation, in the related case record.

When a validation study has been performed for what is most likely to be a one-time analysis, a validated procedure can be applied in casework without the issuance of a standard operating procedure (SOP). In these instances, the following criteria will be met:

- A validation plan will be created and technically reviewed using the appropriate CU *Validation Plan* form and approved prior to commencing validation.
- Step-by-step instructions for the analysis and a summary of the validation performed will be prepared and retained with the validation records.
- The validation records will be technically reviewed and approved by the UC and appropriate TL or SME (as applicable). This will be recorded on the 7-267 or on the CU *Validation Plan and Review- Physical Properties Only* form.
- A copy of the applicable review form and a copy of the step-by-step instructions will be retained in the case notes for the affected case.
- The validation records will be stored electronically and/or in a central location to include the CU's validation file cabinets or bookshelves.
- If and when the procedure is performed again, a SOP will be issued.

[2] The exception to this rule will be when validating the analysis of an unknown component. In these cases, the 7-267 will not be required and the review of the validation will occur as part of the *Laboratory Report* technical review.

4.3 Validation Summary

A *Validation Summary* form (Appendix E) will be completed for each CU validation study that results in a new CU SOP. The individual that led the validation study will complete the form and provide it to the applicable TL. If the TL is the "Lead Scientist" for the study, then another internal SME (if available) will review and approve the validation summary. The summary will briefly describe the performance characteristics that were evaluated to include the values that were obtained for the performance characteristics, if applicable. Other details may be included in the summary. An abstract for a scientific article is a basic model that may be considered when composing the summary.

5 Competency Testing on Newly Validated Analytical Procedures

Caseworking personnel must successfully complete a competency test on a newly validated analytical procedure prior to applying the procedure to casework. This test will demonstrate that applicable personnel can accurately perform the procedure. For personnel that were involved in the validation process, the UC and/or appropriate TL may approve the validation work to serve as demonstration of competency. The successful completion of a competency test, or the approval to use validation work as a substitute for a competency test, will be recorded in the employee's Training and Qualification Records binder.

6 Minor Deviations to Previously Validated Procedures

Minor deviations to SOPs in the CU will be considered for approval by the requestor's TL, and approved prior to the minor deviation being employed. If the requestor is a TL, the minor deviation request will be considered for approval by the UC. If necessary, the UC will consult with an internal SME (if available) and both the UC and the SME will record their approval of the minor deviation.

6.1 Minor Deviation Records

All minor deviations to SOPs will be recorded by the applicable TL in a centralized location. The format of the records is left to the discretion of the TL. At a minimum, the records will include the following:

- FBI Laboratory number(s) or batch code(s) [linked to the FBI Laboratory number(s)] associated with the minor deviation
- Date of the minor deviation
- Personnel that performed the minor deviation
- Personnel that approved the minor deviation

- Title of the document (or unique identifier), issue date and/or revision number, and the specific requirement(s) from which a minor deviation is sought
- A statement of the specific deviation

7 References

FBI Laboratory Practices for Validating Chemical Procedures, FBI Laboratory Operations Manual.

FBI Laboratory Practices for Developing Methods and Validating Technical Procedures

LeBeau, M. et al. "Validation Guidelines for Laboratories Performing Forensic Analysis of Chemical Terrorism", *Forensic Science Communications,* 7(2), April 2005.

Peters, F.T. "Bioanalytical Method Validation and its Implications in Forensic and Clinical Toxicology - A Review", *Accred. Qual. Assur.* 7, 2002, 441-449.

Peters, F.T., Drummer, O.H., and Musshoff, F. "Validation of New Methods", *Forensic Science International*, 165(2-3), 2007, 216-224.

Rev. #	Issue Date	History
10	09/13/19	Separated 'method development', 'develop', etc. from 'validation' throughout to account for the requirement to have method development activities approved and recorded; and to clarify that these are separate activities in CU. Removed bullet from section 4.1.1. Revised section 6.1 to align with Level 1 documents. Added Appendix A- CU *Method Development Plan*.
11	07/15/20	Removed Fire Debris exclusion from Scope. Defined "TL" in section 2, used abbreviation throughout. Replaced "CU Chief" with "UC" throughout. Minor edits made in sections 3.1, 3.2, 3.3, 3.4, 4.2, and 6.1 for clarity. Changed "another CU member qualified in the discipline/category of testing" to "another internal subject matter expert" throughout, resulted in deleting previous section 3.2 and merging with previous section 3.3. Footnote 1 edited to include the validation summary. Defined "SOP" in section 4.2, used abbreviation throughout. Deleted second paragraph from section 4.2, which had allowed for use of a validated procedure prior to SOP issuance. Added use of "batch code" and deleted last bullet in section 6.1. Edited all Appendix forms to Times New Roman text. Changed signature line on Appendices A and E to "Technical Approval" (was some form of "Technical Leader" which doesn't allow flexibility).

Approval

Redacted - Signatures on File

Fire Debris Technical Leader: Date: 07/14/2020

General Chemistry Technical Leader: Date: 07/14/2020

Metallurgy Technical Leader: Date: 07/14/2020

Paints and Polymers Technical Leader: Date: 07/14/2020

Toxicology Technical Leader: Date: 07/14/2020

Chemistry Unit Chief: Date: 07/14/2020

QA Approval

Quality Manager: Date: 07/14/2020

Appendix A: Method Development Plan

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Appendix B: Validation Plan for Qualitative Procedures

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Appendix C: Validation Plan for Quantitative Procedures

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Appendix D: Validation Plan and Review for Physical Property Measurements

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Appendix E: Validation Summary

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